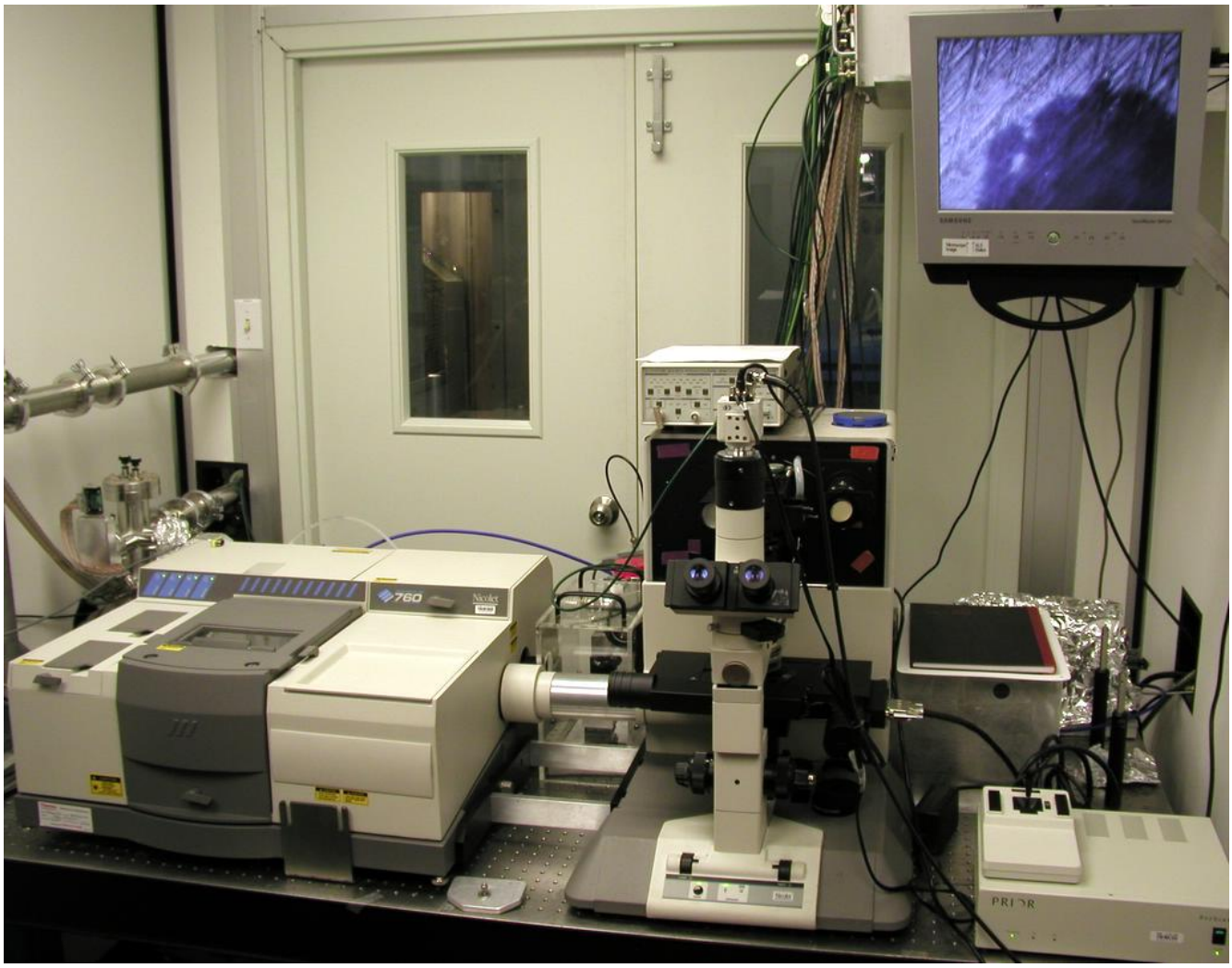


Figure 1: Photograph of the 1.4.3 FTIR spectromicroscopy endstation.

Introduction

The FTIR instrumentation for Beamline 1.4.3 is shown in Figure 1 and consists of a Nicolet 760 FTIR bench
(to the left), a
Spectra-Tech Nic-Plan IR microscope
(in the center), and video display and stage controller for the microscope (on the right). One of the primary reasons for doing infrared spectroscopy at a synchrotron light source is the large enhancement in brightness (flux per unit area). This brightness advantage manifests itself most beneficially when focussing the light to a very small spot size. We have been able to achieve

essentially diffraction-limited spot sizes in the mid-IR using the Nic-Plan microscope with the ALS source.

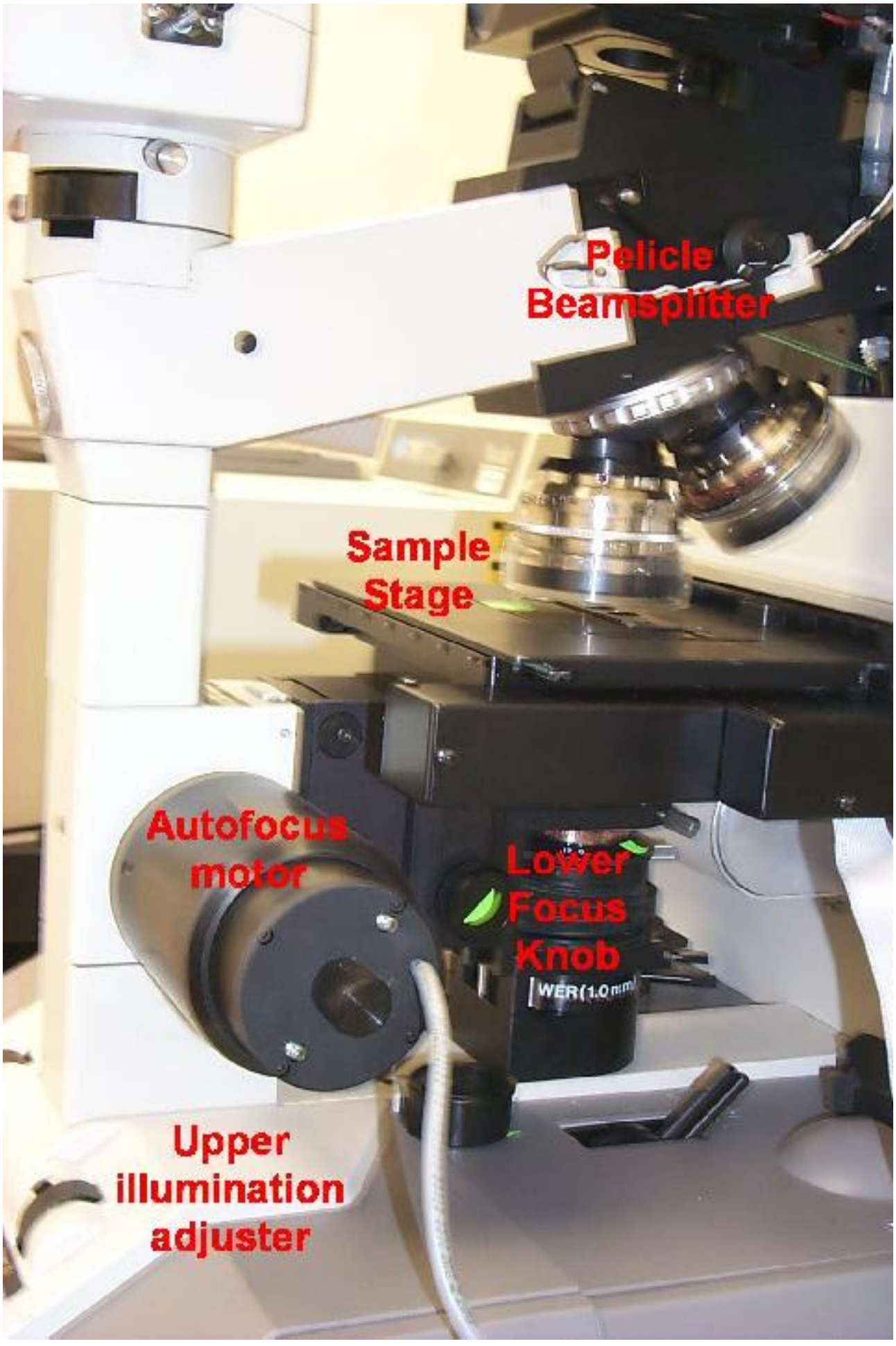
The collimated synchrotron light beam exits the switchyard at its lower left and then enters the beamline 1.4.3 hutch through a hole in the wall. The light is reflected behind the Nicolet 760 bench all in rough vacuum. The beam exits the vacuum via a diamond window and then enters into the side of the Nicolet bench. Once inside the bench, the light goes through the FTIR Michelson interferometer, then is sent on to the sample which can be either in the main bench's sample compartment or in the Nic-Plan IR microscope, and finally to an IR detector. As the microscope is where large advantages are gained from coming to a synchrotron IR beamline, this manual will concentrate on describing the operations of the IR microscope.

When you first arrive at the beamline there are several things to do. The main FTIR bench and the IR microscope are always kept powered on, but the Mercury-Cadmium-Telluride (MCT) IR detector inside the microscope needs to be cooled by filling its dewar with liquid nitrogen. A funnel is built in to the top right of the microscope (gray flip top on a blue plastic mount) and is connected to the MCT's vacuum-insulated dewar. Open the flip top, and slowly pour in liquid nitrogen. A green thermos labeled 'Nicolet' holds approximately the correct amount of liquid nitrogen to fill the MCT detector's dewar. When first beginning to fill, pour in only enough liquid nitrogen to fill the funnel (you will see it start to bubble against the plastic screen), and then stop pouring for at least 60 seconds and let that amount drain down the funnel. Repeat this two or three more times to fully cool down the funnel and tubing system. Then slowly pour the rest of the liquid nitrogen into the funnel making sure to never overflow the funnel. Store the empty green thermos face down on the ground. Please wear safety glasses and cryogenic gloves for this operation. If there is no liquid nitrogen in the 4 L dewar, you may fill it at the user liquid nitrogen station (please see the FAQ for directions). The dewar will typically remain cold for > 10 hours, but you should keep track of when you filled it so that the detector does not get warm over a long measurement!

Setup for Reflection Measurements

Place a reference sample on the microscope stage, for example a gold mirror, and move the x-y stage using the joystick on its controller to place this sample directly below one of the IR microscope objectives. Figure 2 shows a close-up of the sample stage area of the IR

microscope. The microscope has two modes of operation. It can either be in IR mode or View mode, but not both. This ensures that there is never a chance for the alignment laser to be in the microscope when you are looking at a sample with your eyes. Press the ‘view’ button on the front of the microscope to allow visual inspection of the sample, and rotate the upper illumination adjuster to turn on the upper light source. Now look either into the eyepieces or at the TV monitor and adjust the focus (using the coarse and then fine focus knobs on the left of the microscope) looking for the brightest image.


Pelicle
Beamsplitter
Sample
Stage
Autofocus
motor
Lower
Focus
Knob
Upper
illumination
adjuster

Setup for Transmission Measurements

Place your sample on the microscope sample stage over a hole (the black three-holed microscope slides will work for this purpose, or any other holder you have that allows light to pass through the sample). If the sample is transparent to the eye then you can use it to align for transmission mode. If not, place your sample over one hole, and make sure there is another hole available on the sample stage for aligning. Adjust the stage such that the sample is directly below one of the upper microscope objectives and focus on the top surface of your sample, as described in the Reflection measurement setup above.

Once the sample is in focus, we now need to adjust the lower objective's focus to collect the transmitted light from the same location where the upper objective is focussing the incoming light. Insert the circular aperture with a label matching the objective you are using (15x or 32x) into the upper location on the microscope as demonstrated in Figure 4. Lower the black plastic flap in front of the aperture location and slide the appropriate aperture into place taking care to have the tape label on the aperture facing out. This will define a 100-micron area on your sample that we will use to align the lower objective. You should now see the 100-micron area with the rest of your sample masked away on the television monitor or in the microscope eyepieces. DO NOT ADJUST THE MAIN SAMPLE STAGE FOCUS KNOBS ANY FURTHER. If your sample is not visibly transparent, press the SET ORIGIN button twice to memorize the sample position, then move the x-y stage to the open hole using the joystick on the sample stage controller, otherwise just leave the sample where it is and turn down the upper light source intensity.

ill

OMNIC software

e